Sustainable Removal of Ni(II) Ions from Aqueous Solution: Batch Adsorption and Optimization Studies

SHORT SYNOPSIS

Submitted in partial fulfillment of the requirement of the degree of

DOCTOR OF PHILOSOPHY

to

Manav Rachna International Institute of Research and Studies

(Deemed to be University)

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CERTIFICATE

I hereby declare that the short synopsis titled "Sustainable removal of Ni(II) ions from aqueous solution: Batch adsorption and optimization studies" being submitted in partial fulfillment of the requirement for the degree of Doctor of Philosophy in the Department of Applied Sciences (Chemistry under School of Engineering and Technology, of Manav Rachna International Institute of Research & Studies, Faridabad during the academic year 2024-2025. It is a bona fide record of original work carried out under the supervision of Dr. Jyoti Chawla and Dr. Rajeev Kumar and has not been submitted in full or part to any other university or institute for degree or diploma. Further, it is certified that short synopsis is plagiarism free and proof has been submitted.

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ABSTRACT

Nickel (Ni) is a potentially toxic transition element that contaminates soil, air and water. The presence of Ni over permissible limit in environment causes toxicity to all living organisms. Excessive amount of Ni in drinking water may cause health implications like kidney infections, skin allergies, lung fibrosis, cardiovascular issues etc. and also adversely affect the ecological sustainability (Zhang et al., 2019; Gangemi et al., 2009). The aim of current work is to explore/develop the lost-cost materials for the efficient and sustainable removal of Ni(II) ions from water through adsorption. Different characterization tools will be used to validate the applicability of sorbents for the effective removal of Nickel (II) ions from aqueous systems. Response surface method (RSM) will be used to design the experiment and results will be further used for predicting the optimal conditions for maximum adsorptive removal of Ni from water in different conditions. These experimental designs will save the time as well as resources and optimal conditions can be obtained from lesser number of batch experiments. The outcomes of this research will contribute towards sustainable and cost-effective water treatment solutions.

Keywords: Adsorption, Ni(II), optimization, remediation, water treatment

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1. DESCRIPTION OF BROAD AREA

Water contamination by heavy metals has become a global environmental concern, posing serious threats to ecosystems and human health. Among these metals, nickel (Ni) stands out due to its widespread use in industrial processes including metallurgy, electroplating, and battery manufacturing etc. leading to its pervasive presence in water. The environmental implications of nickel contamination range from adverse effects on aquatic life to potential human health risks, emphasizing the urgency of developing strategies for its remediation. Nickel exposure may cause allergic reaction, diarrhea, hepatitis, kidney impairment, gastrointestinal issues, dermatitis and anaemia. Addressing the challenges associated with nickel contamination requires a sustainable framework with a multidisciplinary approach that integrates materials science, environmental engineering, and optimization techniques for removal of nickel ions from water. This research focuses on addressing this challenge through an in-depth investigation into the sustainable removal of Ni(II) ions from aqueous solutions, employing batch adsorption processes and optimization studies.

The proposed study aims to adopt a comprehensive approach by exploring natural/modified novel materials for their adsorption efficiency in removing Ni(II) ions. The selection of adsorbent materials will be done considering the availability, cost-effectiveness, removal efficiency, and eco-friendliness. Standard methods atomic absorption spectrometry/inductively coupled plasma-atomic emission spectroscopy/inductively coupled plasma mass spectrometry will be used to determine the concentration of Ni(II) ions in treated and untreated water samples.

Batch adsorption techniques will be employed to evaluate the performance of these materials under various conditions, including changes in pH, initial nickel ion concentration, contact time, and sorbent dosage. In addition to batch adsorption studies, optimization will be conducted using response surface methodology. The purpose is to fine-tune the removal efficiency of selected materials under various environmental conditions, enhancing the practical applicability of the proposed remediation approach. The overarching goal is to

contribute to sustainable water treatment strategies, providing nuanced insights into the efficient removal of Ni(II) ions while considering environmental protection and resource efficiency which also aligns with sustainable development goal 6 target 6.1.

2. LITERATURE REVIEW

The contamination of water is a global concern since it is a prerequisite for living beings and human livelihood. The rapid rise of the insufficiency of freshwater and its limited availability increases additional environmental issues. The environment has been contaminated with different pollutants such as inorganic, organic pollutants, radioactive isotopes, and gaseous pollutants. Heavy metal pollution is one of the most common environmental contaminants that need extra attention and effective strategies among the different kinds of water pollution due to its toxicity, long residence, non-biodegradable nature, and uncontrolled dispersion. Heavy metals exist naturally on the Earth's crust during the formation of Earth, but anthropogenic activities such as metal mining, using of chemical fertilizers, and industrial manufacturing resulted in an imminent surge of metallic substances in both the terrestrial and the marine environments. The menace of heavy metal contamination in water bodies has spurred extensive research endeavours, and nickel (Ni) ions, as a significant contributor, have garnered considerable attention. Industrial wastewater discharge without treatment may increase the concentration of Nickel in soil/water systems (Babaahmadifooladi et al., 2020).

Nickel (Ni) a ferromagnetic metal is an important biological element for the normal growth of several species of organisms, however, its increased amounts can cause toxic effects such as respiratory disorders, renal inflammation, and extreme generic weakness. Nickel occurs as 5 stable isotopic forms with mass numbers 58, 60, 61, 62 and 64. Nickel 58 is most prevalent (67.8%) followed by 60 (26.2%), 62 (3.7 %), 61 & 64 (1.2%). Nickel may have four oxidation states (I, II, III and IV), and II is its common oxidation state. Salts such as nickel acetate,

nickel chloride, nickel nitrate, and nickel sulfate have good solubility in water in comparison to salts such as nickel carbonate and nickel hydroxide.

Other salts including nickel sulfides, and nickel oxides are nearly insoluble in water. Nickel salts have no distinguishing taste/odour. Nickel salts find extensive applications in batteries, electroplating, and are also used as catalysts. Nickel, a transition metal widely utilized in industrial processes, has permeated aquatic ecosystems, posing environmental and health risks. This widespread use contributes to the increased nickel concentrations in water bodies, making it imperative to explore environmentally friendly and efficient methods for its removal.

In environmental water samples, nickel occurs mainly as $[Ni(H_2O)_6]^{2+}$ in pH range 5 to 9. The maximum permissible concentration of Ni as given by WHO is 2 mg/L in industrial discharge and it should be less than 0.1 mg/L in drinking water. A report by the Central Water Commission, India indicated that nickel concentration varies from 0.00-242.90 µg/L in Indian rivers and maximum nickel was detected at the Elunuthimangalam water quality monitoring station on Noyyal River in 2020. In 199 sites, the concentration of nickel was above the acceptable limits. Studies underscore the need to control Ni(II) ion levels to safeguard ecosystems and human well-being. Electrochemical/membrane-based/adsorption methods may be used for the remediation of nickel ions from aqueous solutions (Kruszelnicka et al., 2022; Kumar et al., 2019).

Commonly used electrochemical techniques are depicted in Figure 1 along with pH conditions. However, these methods are costly due to energy requirements and electrode materials. Membrane-based methods are also widely explored but have certain constraints related to stability, selectivity and efficiency (Rzelewska et al., 2023). Adsorption emerges as a versatile and effective technique for removing Ni(II) ions from aqueous solutions. The rationale behind choosing adsorption is its efficiency, cost-effectiveness, and adaptability to diverse environmental conditions.

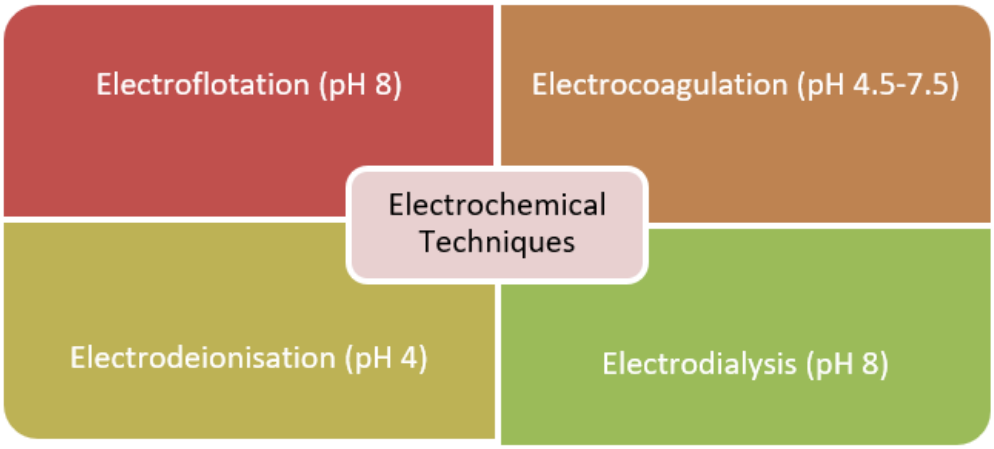


Figure 1: Electrochemical techniques for removal of Ni ions

2.1 ADSORPTION STUDIES

A myriad of materials, both natural and modified, have been investigated for their efficacy in Ni(II) ion adsorption. Agricultural residues, biosorbents, and tailored materials showcase varying degrees of percentage removal. Researchers delve into the intricacies of selecting sorbent materials based on availability, cost, and other related factors. There is a need to explore and utilize the adsorptive materials with a sustainable approach under different conditions. Reported studies (Tables 1 and 2) reveal meticulous investigations into operational variables such as pH, initial Ni(II) concentration, contact time, temperature and adsorbent dosage.

Nickel was adsorbed on tea waste (Malkoc et al., 2005) and reported adsorption capacity (mg/g) was 15.26 at pH 4 and temperature 25 ^{0}C. Shukla et al., 2005 used groundnut shell and saw dust (dose 20 g/L) as an adsorbent for nickel remediation and reported adsorption capacity was 3.83 & 8.05 mg/g respectively at temperatures 35^{0}C. At room temperature and pH 5, watermelon rind (2.5g/L) demonstrated an adsorption capability of 35.3 mg/g.

Alshammari et al., 2020 utilized carob seeds for the removal of nickel from water and reported adsorption capacity (mg/g) was 85.73 at pH 6 and

temperature 25 ^{0}C. Panneerselvam et al., 2021 used adsorbent tobacco dust at pH 4, with an adsorption capacity (mg/g) was 38.3 and a temperature of 25^{0}C.

An illite-smectite clay found naturally in Morocco was studied for its ability to absorb Ni(II) from water. With 206 ppm of Ni(II), 218 mL/min of flow, and 4 g/L of salt, 0.83 mg/g the maximum amount of nickel adsorbed was achieved. The calculated experimental parameters for the maximum repeatability were 108.2 ppm, 94 mL/min, and 20 g/L (Bellido-Milla et al 2023). The adsorption of Ni(II) in aqueous solutions was investigated using synthesized Tpp-MWCNTs (triphenylphosphine-linked multiwalled carbon nanotubes), and their adsorption capacities were contrasted with those of purified MWCNTs. For pure MWCNTs and Tpp-MWCNTs, the adsorption equilibrium was attained in 40 and 30 minutes, respectively. The experimental data was well-fitted by the Freundlich and Langmuir isotherms used to study the adsorption process with high regression coefficient (Adolph et al., 2012).

By using aqueous dispersion and in situ radical polymerization, nanocomposite hydrogels based on poly(methacrylamide-co-acrylic acid) and nano-sized montmorillonite were created. Based on the Langmuir adsorption isotherm, the maximum monolayer adsorption capacity of the nanocomposite hydrogel was determined to be 46.94 mg/g for Ni(II), at 60 min, 6.8 pH, 100 mg/ml adsorbent dose, and 318 K temperature. According to kinetic model pseudo-second order exhibits the highest correlation coefficient and is the greatest fit (Barati et al. 2013). Nettle ash adsorbent was applied for removal of Ni(II) from wastewater. Six was observed to be the ideal pH needed for maximal adsorption. The Freundlich, Langmuir, and Tempkin equations were used to test the experimental data. A monolayer adsorption capacity of 142.8 mg/g was achieved by Langmuir isotherm (Mousavi, & Seyedi, (2011)).

Pigeon peas hulls (Venkata Ramana et al., 2012) were used as sorbent and adsorption capacity was found to be 23.64 mg/g at pH 4 and temperature 40^{0}C. Rafatullah et al., 2009 used Meranti sawdust and reported adsorption capacity was 35.97 mg/g at pH 6 and temperature 30^{0}C. These adsorbents were used at pH less than or equal to 6. Results with other adsorbents used in similar pH conditions are

listed in Table 1. However, Aranda et al., 2018 reported that Quercus crassipes has an adsorption capacity of 104.17 mg/g at pH 8 and temperature of 20 ^{0}C. Gupta et al., 2019 reported that at pH 7 Barbadensis (dose 1g/L) showed a sorption capacity of 10 mg/g at 30^{0}C temperature. Georgieva et al., 2020 in another study used walnut shell (dose 3g/L) as an adsorbent at pH 6.5and the material showed sorption capacity of 13.92 mg/g at a temperature 30^{0}C.

Table 1: Sorption of Ni(II) by natural adsorbents

Adsorbent	**Optimum Conditions**					**Sorbent capacity (mg/g)**	**References**
	pH	**Dose** g/L	**Ni (II) conc. (mg/L)**	**Equi-librium time (min)**	**Temp. (**°C)		
Raw rice bran	5	10	10	60-90	25	NA	Oliveira et al., 2005
Agro-waste of black gram husk	5	10	10	30	25	19.56	Saeed et al., 2005
Tea waste	4	10	NA	120	25	15.26	Malkoc et al., 2005
Groundnut shell	5.16	20	107.75	120	35	3.83	Shukla et al., 2005
Saw dust	5.16	20	107.75	120	35	8.05	Shukla et al., 2005
Watermelon rind	5	2-2.5	50	30	RT	35.3	Lakshmipathy. et al., 2013
Carissa Carandas and Syzygiumaromaticum	6	20	100	180	30	3.76 and 2.96	Sharma et al., 2017
Quercus crassipes	8	1	358	2880	20	104.17	Aranda et al., 2018
Peat	5	5	100	60	30	61.27	Bartczak et al., 2018

Barbadensis Miller leaves	7	1	100	180	30	10	Gupta et al., 2019
Walnut shell	6.5	3	150	120	30	13.92	Georgieva et al., 2020
Groundnut shell	5.16	20	107.75	120	35	7.49	Shukla et al., 2005
Jute fiber	6.58	10	82.3	180	25	5.57	Shukla et al., 2005
Oak sawdust	8	30	97	480	30	3.37	Argun et al., 2007
Tobacco dust	6.34	1	100	90	25	24.5	Qi et al., 2008
Meranti sawdust	6	5	100	120	30	35.97	Rafatullah et al., 2009
Tea leaves	4	6	100	120	40	38.3	Panneerselvam et al., 2011
Tobacco dust	4	6	100	120	40	38.3	Panneerselvam et al., 2011
Pigeon peas hulls	4	0.4	50	40	40	23.64	Venkata Ramana et al., 2012
Maize cob	4	1.25	10	90	RT	NA	Muthusamy et al., 2012
Nigerian-based bamboo	7	10	500	240	25	15-20	Ademiluyi et al., 2016

Carob Seeds	6	1	50	90	25	85.73	Alshammari et al., 2020
Natural Moroccan illite-smectite clay	4.5	20	NA	60	25	0.830	Bellido-Milla et al 2023
Synthesized triphenylphosphinelinked multiwalled carbon nanotubes [TPP-MWCNTs]	6	NA	NA	NA	NA	100	Adolph et al., 2012
Purified MWCNTs	6	NA	NA	NA	NA	55.5	Adolph et al., 2012
Montmorillonite	6	NA	NA	60	25	46	Barati et al., 2013
Nettle ash	6	0.5	50	60	25	192.3	Mousavi et al., 2011
Low cost activated carbon	7	0.05 g/50 mL	50	40	30	43.82	Vijaya kumaran et al., 2009
Modified chitosan	6	0.10 g/100 mL	50	420	25	9.6	Justi et al., 2005
Sodium polyacrylated-grafted AC	NIL	0.10 g/10 mL	NIL	60	25	55.76	Ewecharoen et al., 2009
MWCNTs	NIL	0.05 g/100 mL	80	120	NIL	38.46	Lu and Liu, 2006
Oxidised MWCNTs	6.55	0.015 g/500 mL	20	NIL	NIL	9.8	Chen and Wang, 2006
Purified MWCNTs	6	0.010 g/25 mL	40	30	25	55.55	Muleja et al., 2011
Tpp-MWCNTs (1)	6	0.010	40	30	25	83.33	Muleja et al., 2011

		g/25 mL					
Tpp-MWCNTs (2)	6	0.010 g/25 mL	40	30	25	100	Muleja et al., 2011

***NA -Not available, RT –Room temperature**

Table 1 shows that a wide options of materials have been investigated for the removal of Ni(II) from water and studies confirmed that adsorption capacity is highly dependent on solution pH, initial concentration of Ni(II), and contact time. Various nano adsorbents have shown promising results but there are technical challenges to scaling up their applications as well as environmental concerns and high costs. There is an intense interest in exploring low-cost green alternative materials with promising adsorption and regeneration capacity. The suitability of any material for the removal of nickel can be checked through preliminary adsorption batch studies. However, the performance of adsorptive materials has to be evaluated under different conditions of pH, contact time, adsorbent dose, and initial concentration of ion. Experimental conditions are required to be further optimized and this can be done by taking one variable at a time and keeping others constant. This requires many experiments to be performed to study the effect of all variables. It's time consuming and a large number of reagents are required to study the interaction effects among the affecting variables. This can be solved by utilizing multi-variate approach for assessment of interaction of affecting factors on the adsorption process and to get optimal conditions for better removal.

2.2 OPTIMIZATION OF ADSORPTION PROCESS

Different approaches of response surface methodology (RSM) have been used for the optimization of adsorption process. The response surface methodology includes different techniques that can be applied for the assessment of the association between a set of controlled experimental factors and measured responses based on different conditions. Biswas et al., 2019 utilized a biochar biopolymeric hybrid adsorbent for the removal of Ni(II) from water and optimized the removal process using response surface methodology. The model fitted well and had good agreement with the experimental

observations. The reported adsorption capacity was 156 mg/g. Ince et al., 2022 used bio-composite material for Ni(II) ions from industrial wastewater and applied a central composite design to predict the performance of the material. The adsorption capacity was reported as 6.28 mg/g and the maximum adsorption capacity was predicted at 6.9 pH, 150.1 mg/L of sorbent, and 39.7 mg/L of initial metal ion concentration. Garg et al., 2008 studied nickel removal from water using sugarcane bagasse and studied the effect of three conditions including adsorbent dose, pH and stirring speed on the removal of nickel using central composite face-centered design. Ebrahim et al., 2020 investigated the performance of coal-based sorbent using response surface methodology and predicted 73% removal of nickel ions under selected conditions. Nwafulugo et al., 2014 applied RSM for the optimization of the removal of nickel from wastewater successfully and obtained the optimum conditions of pH, contact time and concentration of sorbent material for maximum removal. RSM three-level Box–Behnken design (BBD) has also been found to be useful in the flow electrode capacitive deionization process for nickel remediation (Pelarti et al., 2022). RSM was also employed (Babakhani & Sartaj, 2023) to get an idea about a correlation between pH, chitosan crosslinking degree, and the Ni(II) adsorption capacity and suggested model was in good agreement with the experimental data. The RSM based on central composite design (CCD) approach was also used to understand the effects of the operating variables on the adsorption of nickel (Kolbadinejad & Ghaemi, 2024).

These techniques have been successfully used to examine the effects of various experimental conditions, and interactive effects of effecting variables, and predict the optimum conditions for desired responses. Only few studies have been reported that include the application of response surface methodology to predict the optimum combination of studied variables in different conditions for the removal of nickel from water. A key aspect of current research is to investigate the performance/feasibility of using various materials for the removal of nickel ions from water based on their adsorption capacity, regeneration capabilities, cost considerations, and accessibility. RSM studies will be helpful to select the optimum conditions for removal as per the nature of sample to be treated.

3. RESEARCH GAPS

In summary, the literature review underscores the urgency and significance of exploring novel sustainable approach for the remediation of nickel from water. The performance of material as adsorbent for removal of Ni(II) from water in different conditions need to be analyzed considering sustainability factors, cost & availability of material, adsorption and regeneration capacity. A sustainable framework with minimum number of experimental runs needs to be designed with the help of preliminary batch adsorption experiments and advanced computational/ mathematical techniques that can reduce the time as well as overall cost required for adsorption experiments. This multi-variate approach can also benefit in assessment of interaction of affecting factors on adsorption process and to predict optimal conditions for better removal.

4. PROBLEM STATEMENT

To explore/prepare low cost, efficient and ecofriendly sorbents for removal of Ni(II) ions from water and to develop a sustainable framework using response surface methodology. The overarching requirement is to strike a balance between removal effectiveness and environmental responsibility, thereby fostering the development of resource-efficient and eco-friendly approaches for sustainable Ni(II) ion removal from water.

5. RESEARCH OBJECTIVES

The research aims to contribute substantively to the field of water treatment, offering practical solutions for sustainable removal of Ni(II) ions from aqueous solutions. The specific objectives are outlined as follows:

1. To explore the natural materials for removal of Ni(II) ions from water and examine the feasibility of facile modifications of natural materials for better efficiency as adsorbents.
2. To employ advanced analytical techniques such as Fourier transform infrared spectroscopy (FTIR), energy dispersive X-ray spectrometer (EDX), scanning electron microscopy (SEM), and X-ray diffraction (XRD) to comprehensively characterize both natural and modified materials.
3. To conduct batch studies to analyze the impact of crucial operational variables, including initial Ni(II) concentration, pH, contact time, and adsorbent dosage, on the

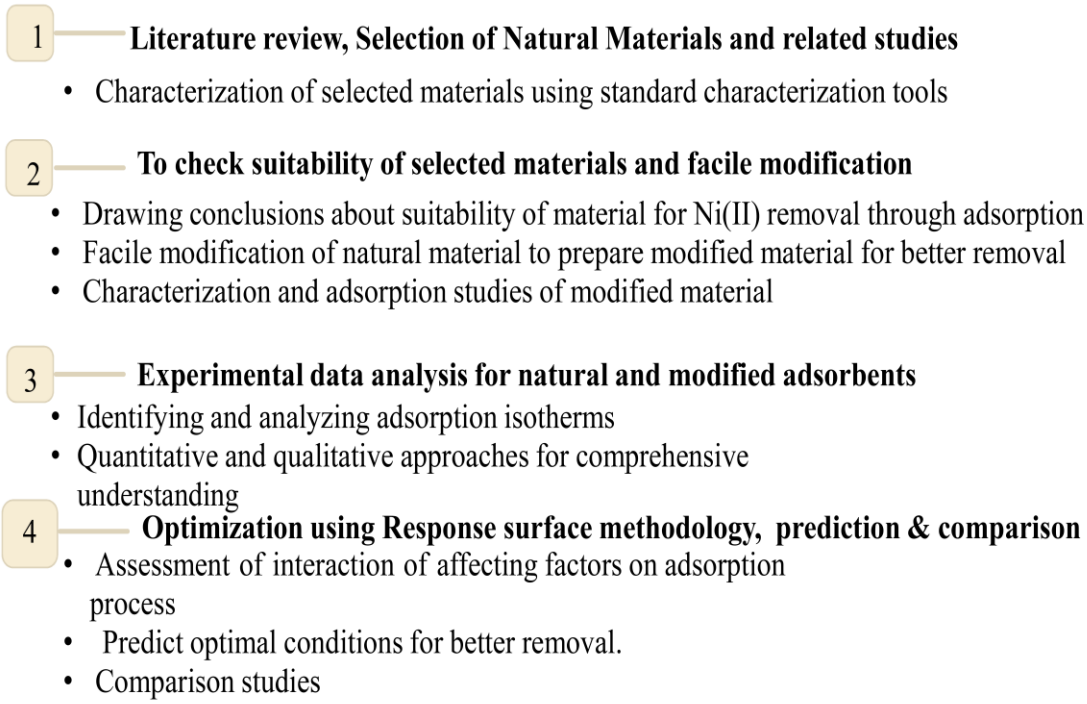
removal efficiency of natural and modified materials. Standard methods will be used to determine the concentration of Ni(II) ions before and after using sorbent material.

4. Adsorption isotherm studies to elucidate the type and mechanism of Ni(II) ion adsorption.

5. To utilize Response surface methodology to optimize the removal efficiency of selected materials under diverse conditions, focusing on achieving maximum efficacy in Ni(II) ion removal. Design Expert software will be used to design the adsorption experiments called runs, in which changes will be made in the pH, adsorbent dose, and contact time as input variables in order to identify the changes in removal percentage.

6. To compare the performance of the identified materials with other potential adsorbents, considering various factors.

6. RESEARCH METHODOLOGY


1
Literature review, Selection of Natural Materials and related studies
• Characterization of selected materials using standard characterization tools
2
To check suitability of selected materials and facile modification
• Drawing conclusions about suitability of material for Ni(II) removal through adsorption
• Facile modification of natural material to prepare modified material for better removal
• Characterization and adsorption studies of modified material
3
Experimental data analysis for natural and modified adsorbents
• Identifying and analyzing adsorption isotherms
• Quantitative and qualitative approaches for comprehensive understanding
4
Optimization using Response surface methodology, prediction & comparison
• Assessment of interaction of affecting factors on adsorption process
• Predict optimal conditions for better removal.
• Comparison studies

7. EXPECTED OUTCOME OF THE RESEARCH

1. Development of green novel material for removal of Ni(II) ions from water.
2. The performance of material will be determined with the help of adsorption batch studies. The results may be used for practical recommendations for water treatment based on the findings of adsorption studies.
3. Adsorption efficiency of materials can be predicted in different conditions of treatment with the help of RSM.
4. Contribution to the development of practical and sustainable water treatment strategies for Ni(II) ion removal.
5. The research work will provide a comprehensive sustainable framework for removal of Ni(II) ions from water ensuring a thorough and impactful analysis of planned adsorption experiments.

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